

**CORRELATION OF CREEP RATE WITH MICROSTRUCTURAL CHANGES
DURING HIGH TEMPERATURE CREEP**

NASA-CR-135698) CORRELATION OF CREEP
RATE WITH MICROSTRUCTURAL CHANGES DURING
HIGH TEMPERATURE CREEP Annual Report, 1
Jul. 1972 - (Virginia Polytechnic Inst.
and State Univ.) 37 p HC \$4.00 CSCL 11F

N73-32426

Unclas
G3/17 15740

Annual Report

Metallurgical Engineering
Division of Minerals Engineering
Virginia Polytechnic Institute and State University

Period: July 1, 1972 to June 30, 1973

NASA Grant NGR 47-004-108

September, 1973

by

C. T. Young

W. A. Hochella

J. L. Lytton

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I. ABSTRACT

In future designs of subsonic and hypersonic aircraft and space vehicles, many structural metallic components will be designed to withstand repeated applications of complex stress cycles at elevated temperatures and will generally be required to experience less than 0.5% creep strain during the design life. Consequently, the primary stage of creep is of significant importance. It is known that the course of primary creep can be favorably altered by the use of various thermomechanical treatments to produce more creep-resistant substructures, but our understanding of how the stability of these structures during primary creep is related to creep rate is limited.

In this research the techniques of electron microscopy are being used to examine the microstructural changes which occur during primary creep for two important types of engineering alloys: (1) alloys strengthened by solid-solution additions, and (2) dispersion-strengthened alloys. The metals chosen for study are unalloyed titanium, Ti-6Al-4V, and the cobalt-base alloy, Haynes 188.

Results to date on NGR 47-004-108 show that development of prior dislocation substructure in Haynes 188 by 10% prestrain and annealing for one hour at 1800°F increases the time to reach 0.5% creep strain at 1600°F by more than an order of magnitude for creep stresses from 3 to 20 ksi. For creep at 1800°F, similar results were obtained for stresses above 7 ksi, but the prior substructure decreases creep resistance below 7 ksi. This effect appears to be related to instability of grain structure at 1800°F in prestrained material.

In future work, creep tests and electron microscopy will be used to study the effects of prior substructure on the primary creep behavior of unalloyed titanium and Ti-6Al-4V. Further work on Haynes 188 will be performed to relate dislocation and carbide morphologies to the creep results obtained.

II. INTRODUCTION

The effects of the external variables of stress and temperature on the steady-state creep behavior of pure metals and dilute alloys at elevated temperatures has been extensively studied and an understanding of these effects is gradually emerging⁽¹⁻³⁾. For example, the temperature dependence of creep is now believed to be simply related to self-diffusion and the effect of stress on creep rate can usually be expressed as a power law of exponent 4-6 for the normal range of stresses encountered in engineering service. Recently, the effects of the physical parameters of elastic modulus and stacking fault energy have also been included in creep correlations^(1,2).

The important effects of microstructure on creep behavior, however, are still not well understood. For example, the initial creep rate of high purity aluminum may be decreased by more than four orders of magnitude, for the same stress and temperature, simply by changing the initial microstructure from the as-annealed state to a structure containing subgrains bounded by dislocation networks formed by prior thermomechanical treatments^(4,5). In these studies, the microstructural changes were studied by transmission electron microscopy, and it was found that the average subgrain sizes developed by 5% and 10% prestrain and recovery treatments were 2.51 microns and 1.75 microns respectively. After creep, the sizes were found to be 3.87 and 3.41 microns respectively, illustrating the tendency for development of substructures during primary creep which are in equilibrium with the applied stress. It has been generally accepted that smaller subgrains are more creep resistant^(1,2), but, for the specimen

given 5% prestrain, the creep rate decreased by a factor of ten while the subgrain size increased. It must be concluded that the subboundaries can become more creep resistant as they move further apart during primary creep. It is believed by some authors that changes in subboundary misorientation angle are probably not important during steady-state creep⁽²⁾, but it is clear that any meaningful study of primary creep must examine detailed nature of dislocation subboundaries as well as their spacing. In order to facilitate the measurement of subboundary angles using Kikuchi patterns, Young and Lytton⁽⁶⁾ and Young, et al.⁽⁷⁾ have recently developed computer programs to provide for more rapid and accurate determination of subgrain misorientation angles.

In future designs of subsonic and hypersonic aircraft and space vehicles, many structural metallic components will be designed to withstand repeated applications of complex stress cycles at elevated temperatures during operation at altitudes ranging from sea level to more than 400,000 feet. Since these metals will generally be required to experience less than 0.5% creep strain during their design life, it is clear that significant attention needs to be given to the microstructural changes which occur during primary creep, since 0.5% creep strain generally falls within the primary stage.

It is therefore anticipated that significant improvement of design life can be obtained in high temperature alloys by development of improved initial dislocation and precipitate structures through appropriate thermomechanical treatments.

The purpose of the proposed research is to examine the relation of creep rate to microstructure during primary creep for two important classes of high temperature alloys: (1) alloys strengthened by solid solution

additions, and (2) alloys which can be strengthened by dispersed phases. The metals and alloys chosen for the present study were unalloyed titanium, Ti-6Al-4V, and the cobalt-base alloy, Haynes 188. The study of titanium and Ti-6Al-4V is intended to illucidate the important relationship between creep rate and dislocation structures formed by prior thermomechanical treatments with special reference to how these are affected by solute additions.

Study of the creep behavior of Haynes 188 is intended to reveal how the superimposed effects of dislocation structure and finely dispersed precipitates are related to creep rate and microstructural stability. Variations in creep rate for Al-0.5 at % Ag have recently been related to the effects of pinning of dislocation substructure by γ' precipitates and their gradual growth during creep straining, and these effects apparently account for unusual temperature and stress dependence of the creep rate^(8,9). In the case of Haynes 188, the dispersed phases available are predominantly two types of carbides, M_6C and $M_{23}C_6$. The matrix is stabilized in the FCC structure by nickel, iron, carbon, and manganese^(10,11). The M_6C carbides are found dispersed in the matrix after annealing at $2150^{\circ}F$, and further exposure at temperatures above about $1650^{\circ}F$ tends to produce additional precipitation of M_6C . Exposure between 1300° and $1650^{\circ}F$, however, tends to produce the complex chromium-nickel-cobalt carbide, $M_{23}C_6$. This $M_{23}C_6$ carbide appears to be heterogeneously nucleated, and offers significant promise for improved creep strength during primary creep by interaction with dislocation structures produced by thermomechanical means.

The description of results which follows represents work accomplished during the first 12 months of NASA Grant NGR 47-004-108 which was funded for the

15-month period beginning July 1, 1972. It is anticipated that present funds will be expended by October 30, 1973.

III. RESULTS TO DATE

The investigation related to the enhancement of creep resistance of Haynes 188 cobalt-base alloy by thermomechanical treatment has been conducted at Langley Research Center. The study concerning the effect of thermomechanical treatment on the creep behavior of pure titanium has been carried out at VPI&SU. In addition, some significant improvements have been made in the computer program for producing Kikuchi patterns which is to be used in the study of subgrain boundaries. The results to date in these three areas are presented in this section.

Haynes 188 Cobalt-Base Alloy

The material used in this study was obtained from the Stellite Division of the Cabot Corporation. The nominal composition of this alloy is 20-24% Cr, 20-24% Ni, 13-16% W, 0.05-0.15% C, 0.03-0.15% La, 0.20-0.50% Si, 1.25% Mn, up to 3% Fe, with the balance cobalt. The sheet was received in the mill annealed condition and had been stretch flattened 1-2%. The 0.2% offset yield strength was found to average about 78 ksi, compared with that specified by the producer to be 67 ksi for annealed sheet. It is believed that this difference is due to the cold work produced during stretch flattening. Creep specimens were machined into the configuration shown in Figure 1. Some of the specimens were strained to 10% true tensile strain using an Instron machine. In the present study, creep specimens were given a preoxidation treatment in air at 1800°F for one hour. The primary purpose of this preoxidation treatment was to form a layer of oxide which provides for high surface emissivity, a condition required for the material to be used as a thermal

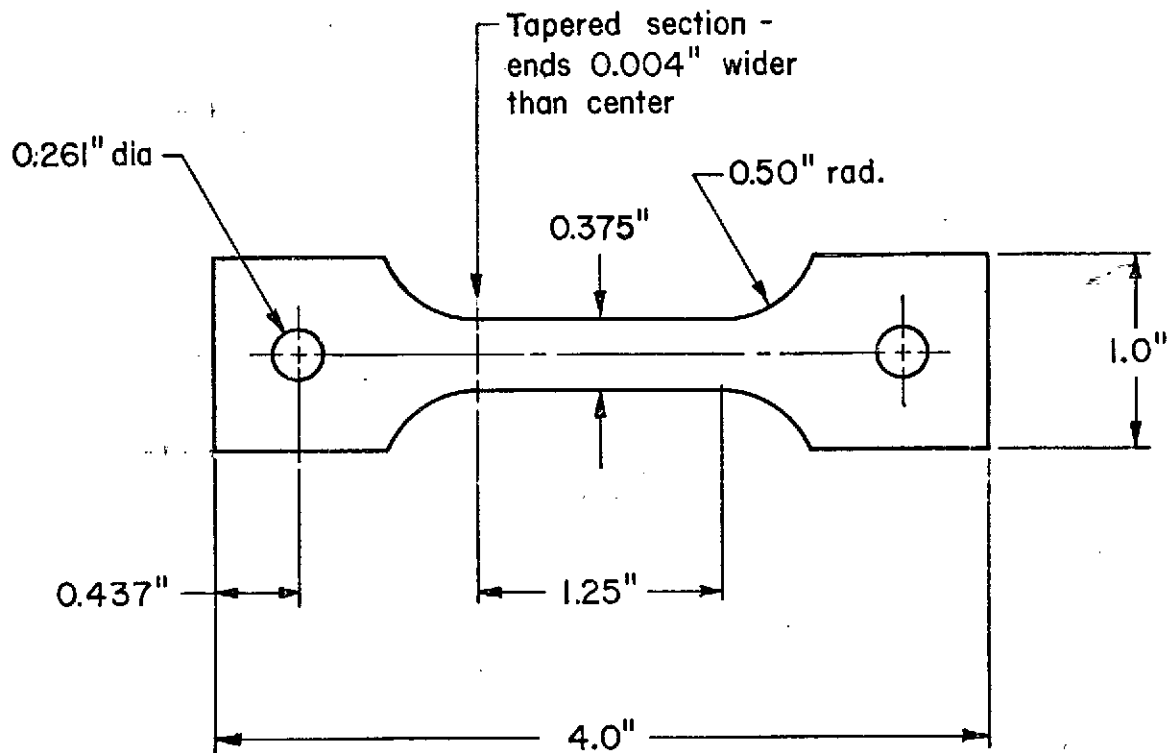


Figure 1. Creep and tensile test specimen configuration.

protection shield in certain space applications. Creep specimens were placed in the creep furnace and three to four hours were allowed for reaching the equilibrium temperature desired for creep testing.

Flow Stress Recovery After Cold Work

In view of the various thermomechanical treatments which were to be used to generate stable substructures prior to creep tests, an investigation was made to determine if the one hour preoxidation treatment at 1800°F also could serve as a flow stress recovery treatment and provide a nearly stable dislocation structure when conducted following the 10% prestrain. For this purpose, the room temperature 0.2% offset yield strength of the alloy was measured after various recovery times at 1800°F in air and the results are shown in Figure 2. It is seen that the flow stress of the as-received alloy drops from 78 ksi to 64 ksi within one hour of annealing and stays at this value for at least 24 hours. The flow stress of the specimens prestrained 10% drops from 115 ksi to about 90 ksi within one hour and decreases slowly on further exposure to about 85 ksi in 24 hours. Apparently, age hardening does not occur for either the as-received or the prestrained alloys, at least for the first 24 hours. The decrease in yield strength during annealing is believed to be due to the rearrangement of dislocations which were introduced during cold working. The retention of increased yield strength of the prestrained alloy after the 24 hour preoxidation treatment is believed to be due to the metastable dislocation structures developed and perhaps to a favorable distribution of precipitate particles. It has been concluded that the one hour preoxidation treatment at 1800°F is sufficient to produce a nearly stable dislocation substructure following 10% room temperature prestrain in Haynes 188.

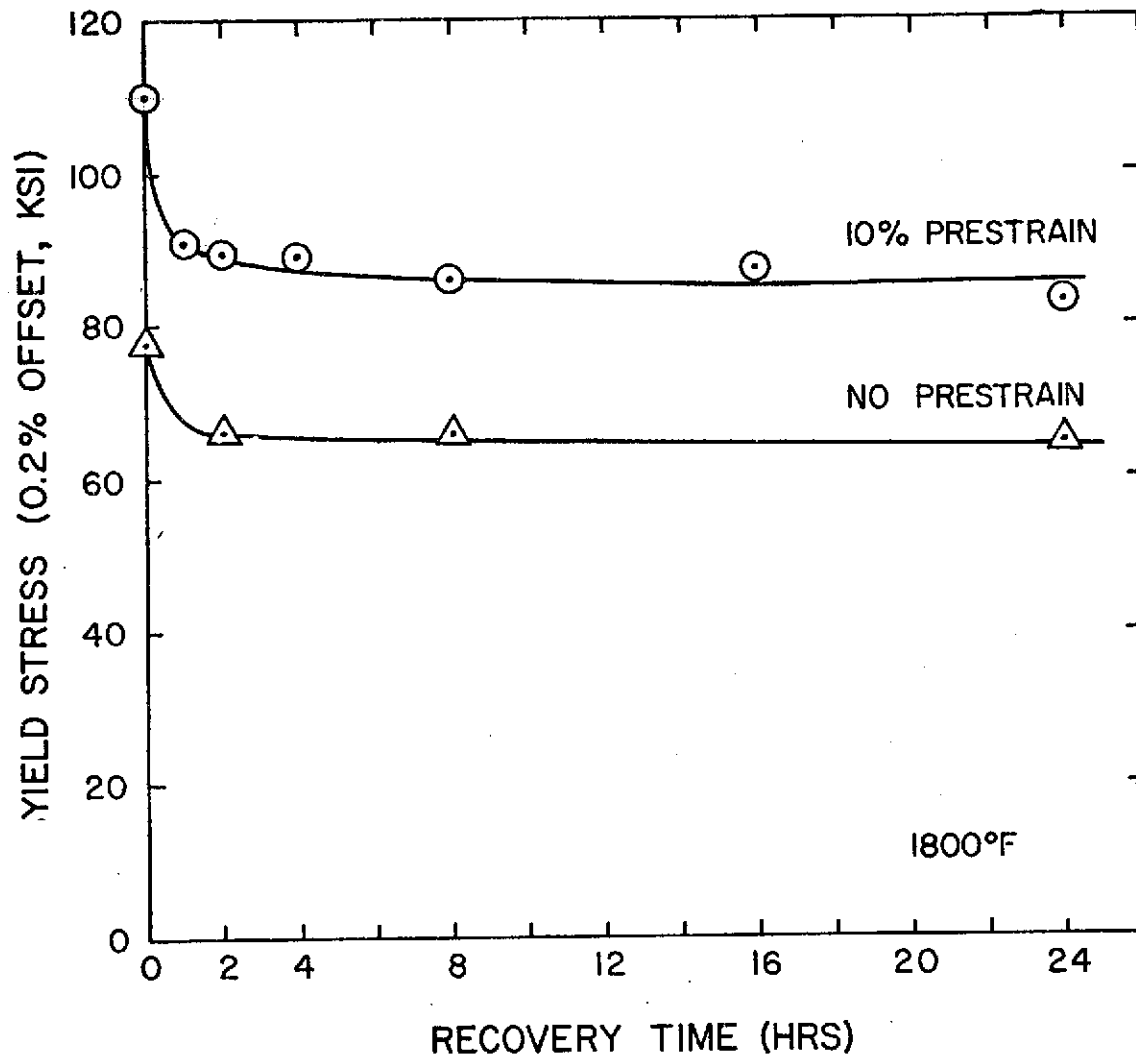


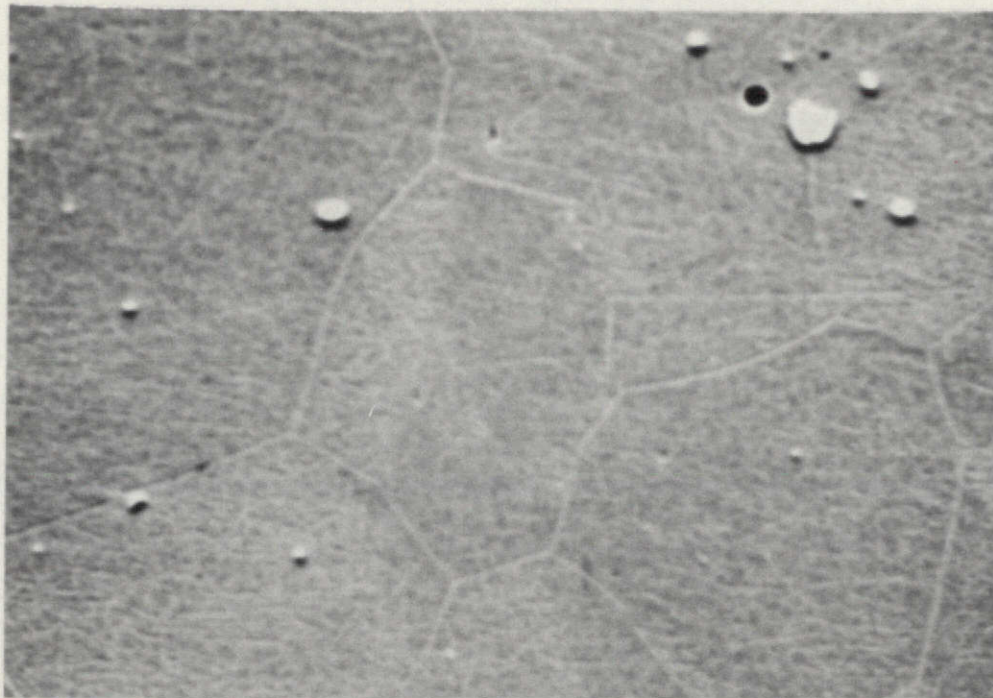
Figure 2. Flow stress recovery behavior of Haynes 188 at 1800°F.

Scanning electron micrographs at 1000X of polished and etched specimens of Haynes 188 are shown in Figure 3. Figure 3a shows the microstructure of the as-received, mill annealed, sheet; and Figure 3b shows the structure after 10% prestrain and the one hour preoxidation treatment at 1800°F. Note that the as-received sheet has no visible grain boundary precipitates; the white precipitates are reported to be M_6C carbides⁽¹¹⁾. In the prestrained and preoxidized material, however, the grain boundaries and many twin boundaries are now seen to be decorated with precipitate. In view of established results of aging studies at 1800°F⁽¹¹⁾, as shown in Figure 4, it is believed that these precipitates are also of the M_6C type, since aging one hour at 1800°F remains within the M_6C field. The grain size of the Haynes 188 alloy was found to be about 0.03 mm.

Creep Behavior of Haynes 188

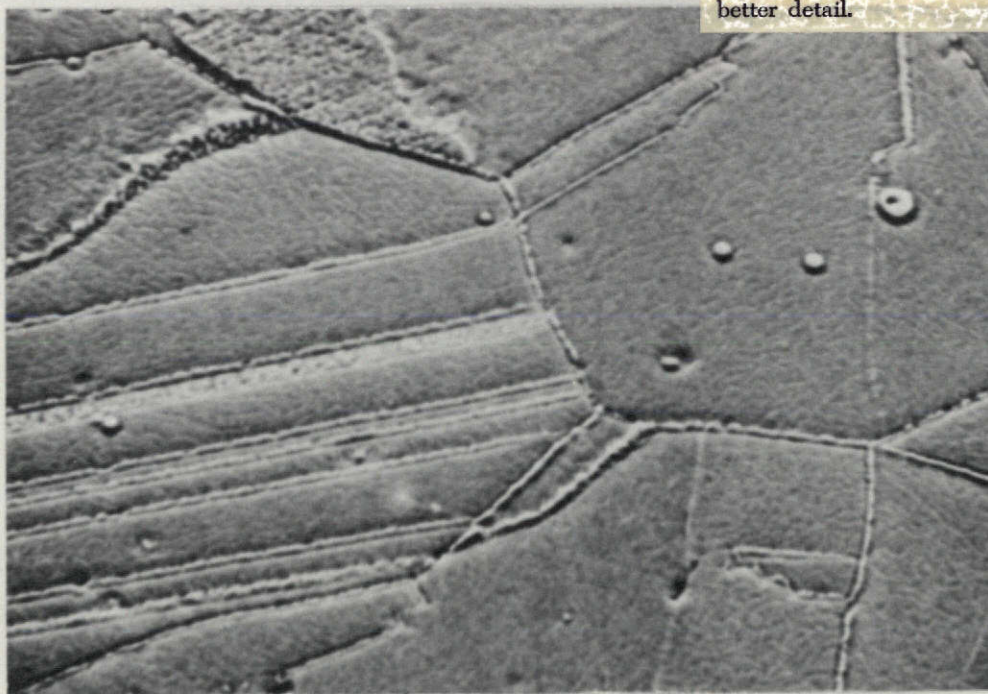
Constant load creep tests were conducted at a pressure of 1 torr and at temperatures of 1600°F (871°C) and 1800°F (972°C). Stresses employed to date range from 3 ksi to 20 ksi. The temperature was maintained to $\pm 3^\circ\text{C}$. A platinum strip strain gauge was attached to each specimen and a microscope equipped with a vernier micrometer was used to read the creep strains. The creep strain so determined is accurate to ± 0.0003 . The thermomechanical treatments prior to creep testing and the creep test parameters that have been employed to date are summarized in Table I.

The various types of creep curves which have been obtained thus far are shown in Figures 5, 6, and 7. For creep at 1800°F and 15 ksi (Fig. 5) it should be noted that the thermomechanically treated specimen had a minimum creep rate which was ten times less than that of the as-received sheet. The strain at creep rupture, however, also decreased from 0.285



(a) Mill annealed at 2150°F, stretch flattened.

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(b) 10% prestrained, preoxidized one hour at 1800°F.

Figure 3. Scanning electron micrographs of polished etched Haynes 188 alloy. 1500X.

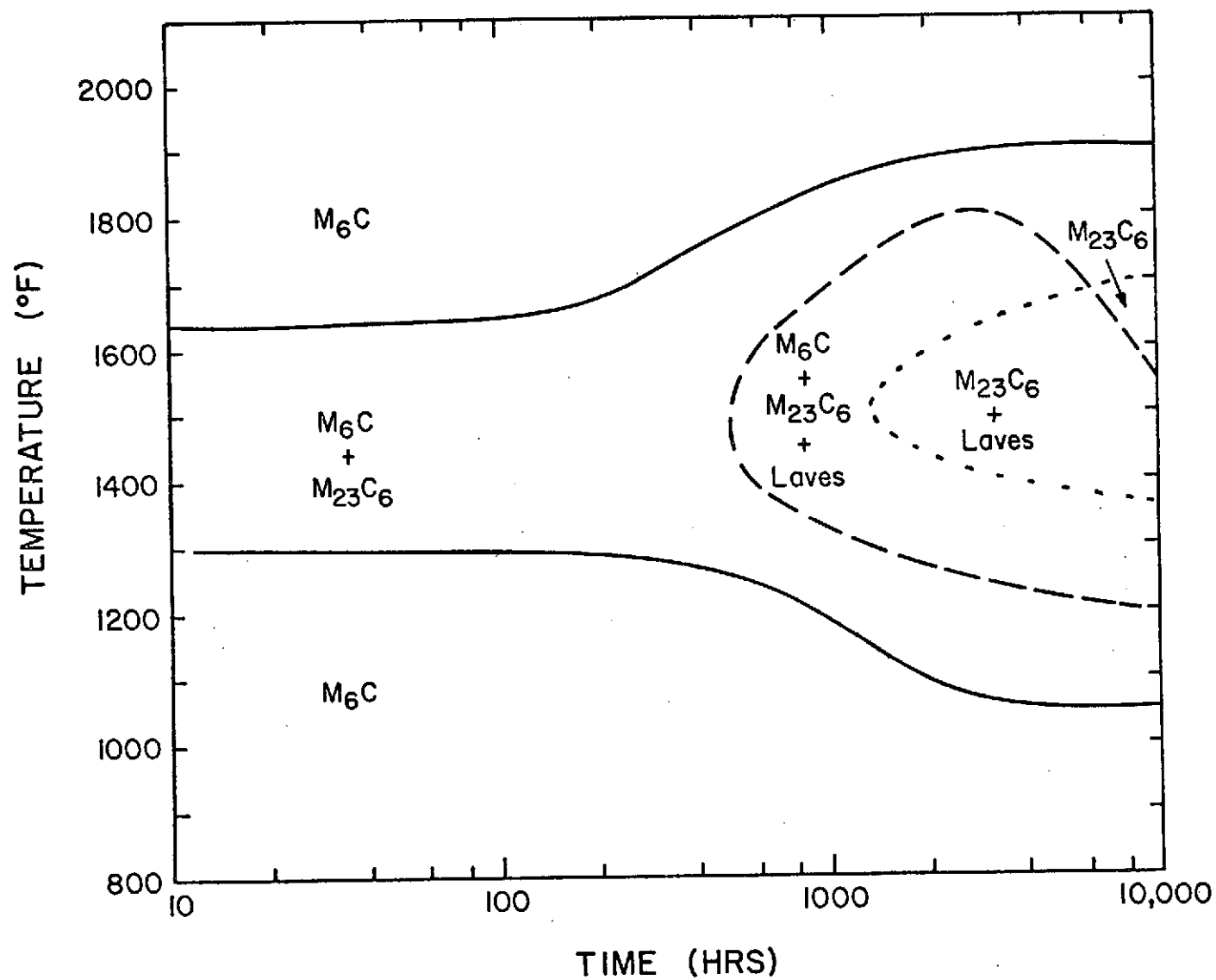


Figure 4. Structural reactions during aging of Haynes 188. Order of phases listed indicates relative abundance. (After Herchenroeder, Ref. 11)

Table I - Haynes 188 Creep Test Conditions

<u>Thermomechanical Treatment</u>	<u>Creep Parameters</u>	
	<u>Temp. (^oF)</u>	<u>Stress (ksi)</u>
AR* + 1800 ^o F/1 hr/air	1600	5,10,15,20
	1800	3,5,10,15
AR + 10% _e + 1800 ^o F/1 hr/air	1600	5,10,15,20
	1800	3,5,10,15
AR	1600	15
AR + 10% _e	1600	15

*AR - as-received (1-2% strained)

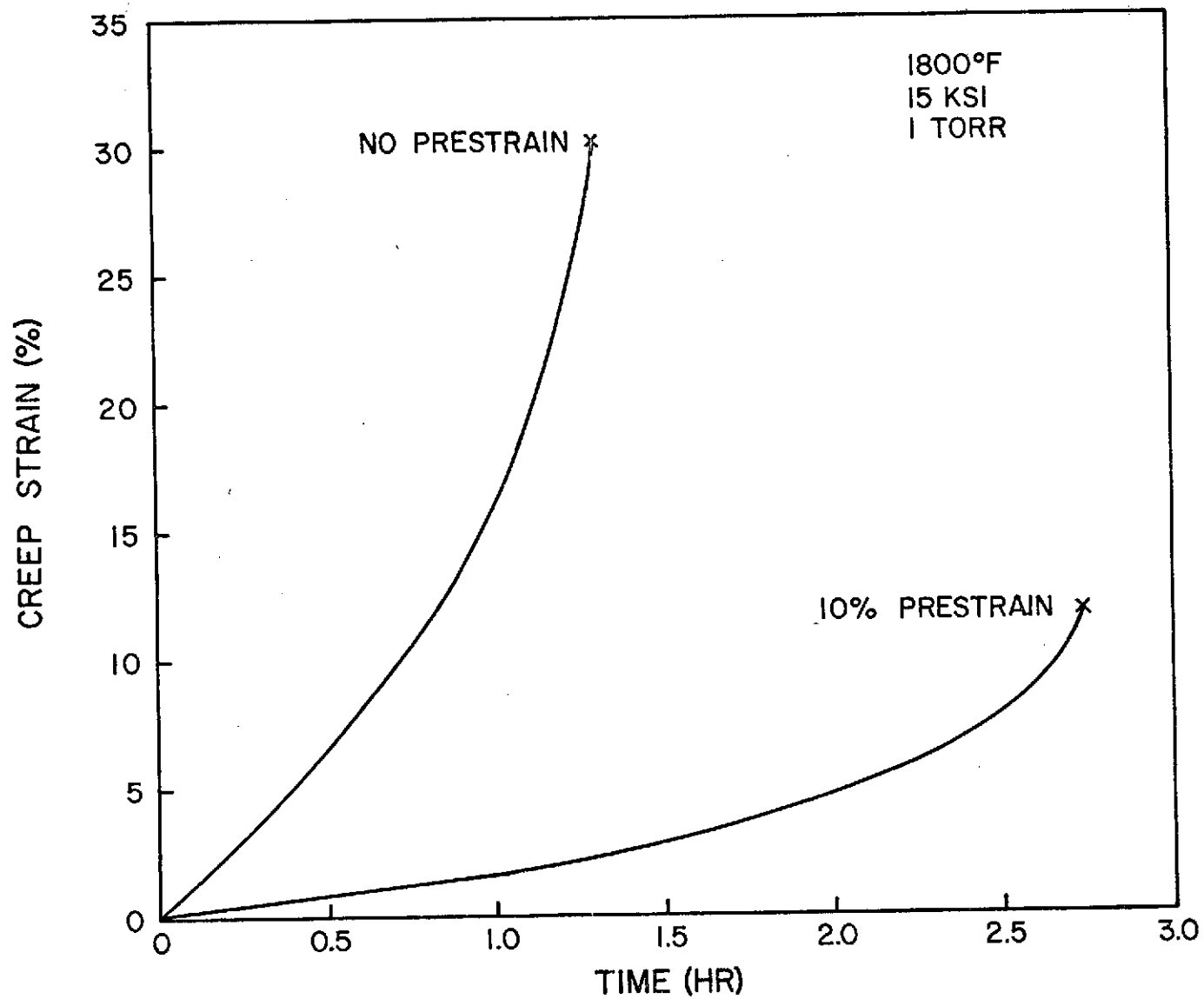


Figure 5. Creep curves for Haynes 188 alloy at 1800°F and 15 ksi.

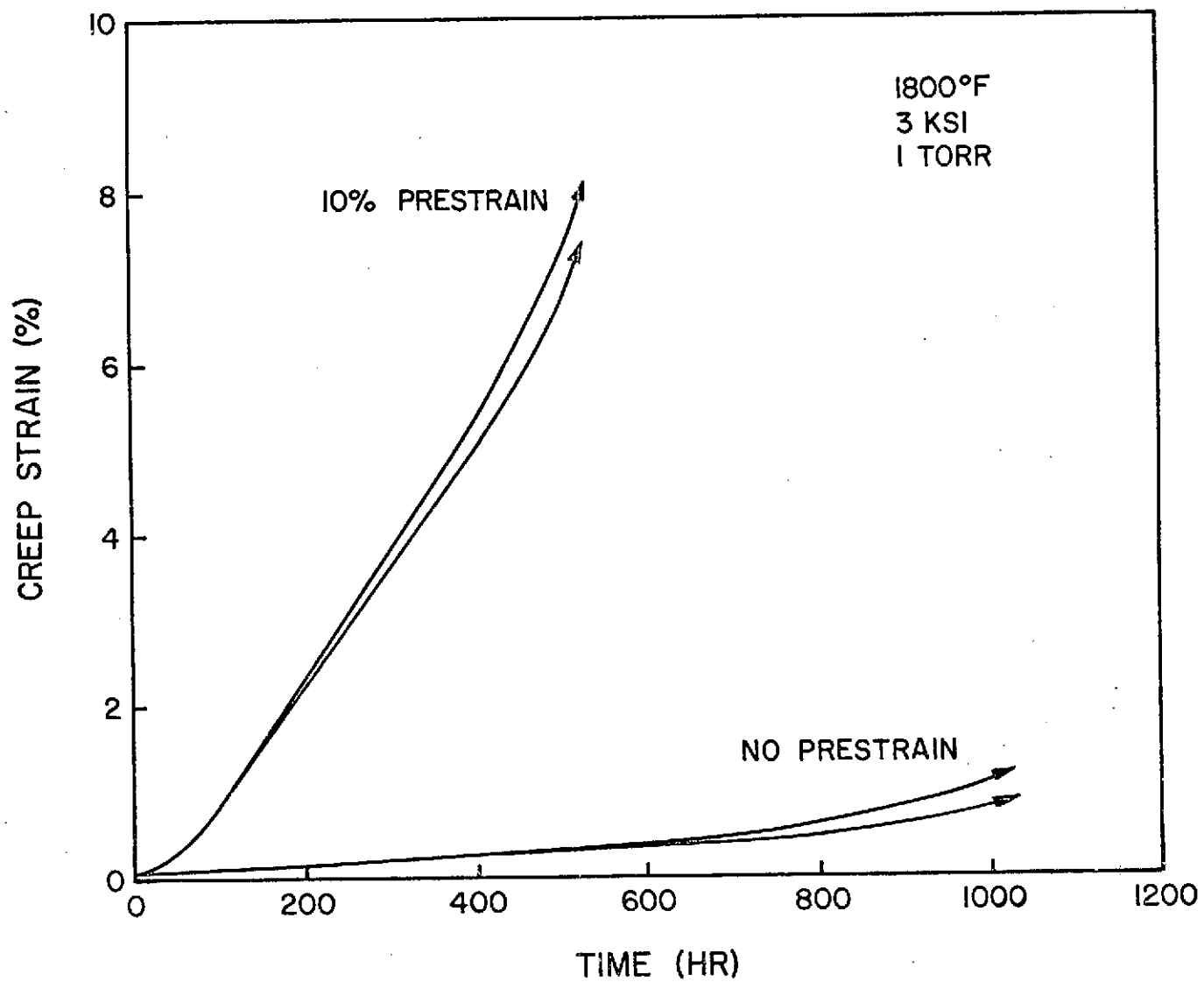


Figure 6. Creep curves for Haynes 188 alloy at 1800°F and 3 ksi.

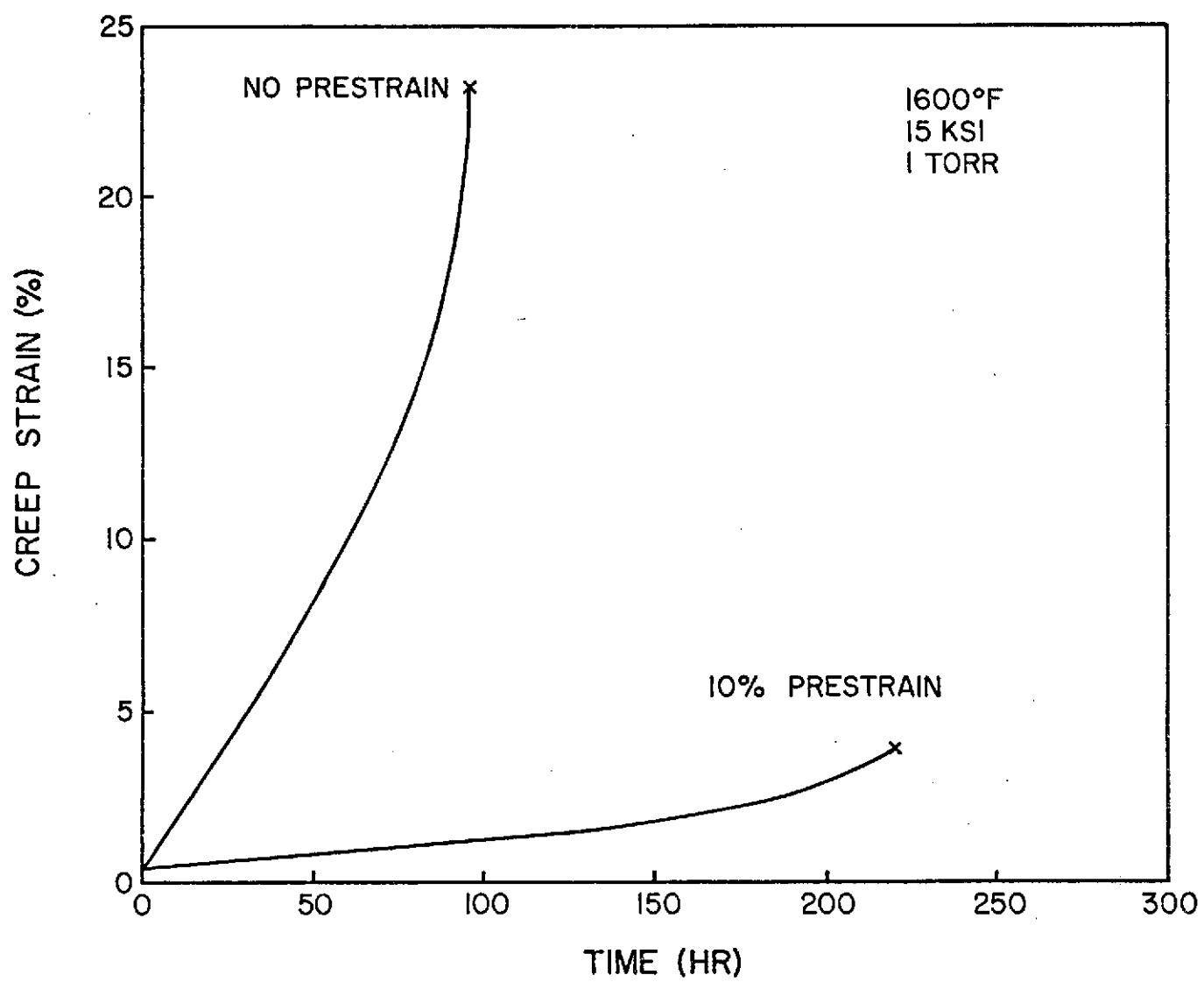


Figure 7. Creep curves for Haynes 188 alloy at 1600°F and 15 ksi.

to 0.117. At 1800°F and 3 ksi the as-received specimen had a minimum creep rate which was nine times less than for the specimen prestrained 10% (see Figure 6). The tests at 3 ksi were discontinued prior to fracture.

At 1600°F no loss of the beneficial effects of prior thermomechanical treatment was found. A typical creep curve for 1600°F and 15 ksi is shown in Figure 7. Under these conditions, the thermomechanically treated specimen had a minimum creep rate which was about 15 times less than the as-received, mill annealed sheet. This beneficial effect was found at all stresses at 1600°F, as illustrated in Figure 8 which shows the minimum creep rates as a function of creep stress. For comparison, the times to reach 0.5% creep strain as a function of creep stress for the various tests are shown in Figure 9. The times greater than 2000 hours were estimated by extrapolation from the creep curves.

The present results, summarized in Figures 8 and 9, show that, for creep at 1800°F, the beneficial effects of the prior substructure are limited to stresses above about 7 ksi. At 1600°F, however, the 10% prestrain and 1800°F anneal has been found to decrease the minimum creep rate by a factor of ten or more, with this factor apparently increasing somewhat with decreasing stress. It appears clear that the substructural effects produced during creep at 1600°F are markedly different from those at 1800°F.

While a detailed explanation of the inverse effect of the prestrain-recovery treatment for stresses below 7 ksi is not yet available, it is reasonable to suspect that some form of structural instability occurs during long-time exposure at 1800°F when prior straining has been performed. Reference to Figure 4 shows that the appearance of the $M_{23}C_6$ carbide is to be expected at about 500 hours static aging at 1800°F. Under dynamic conditions

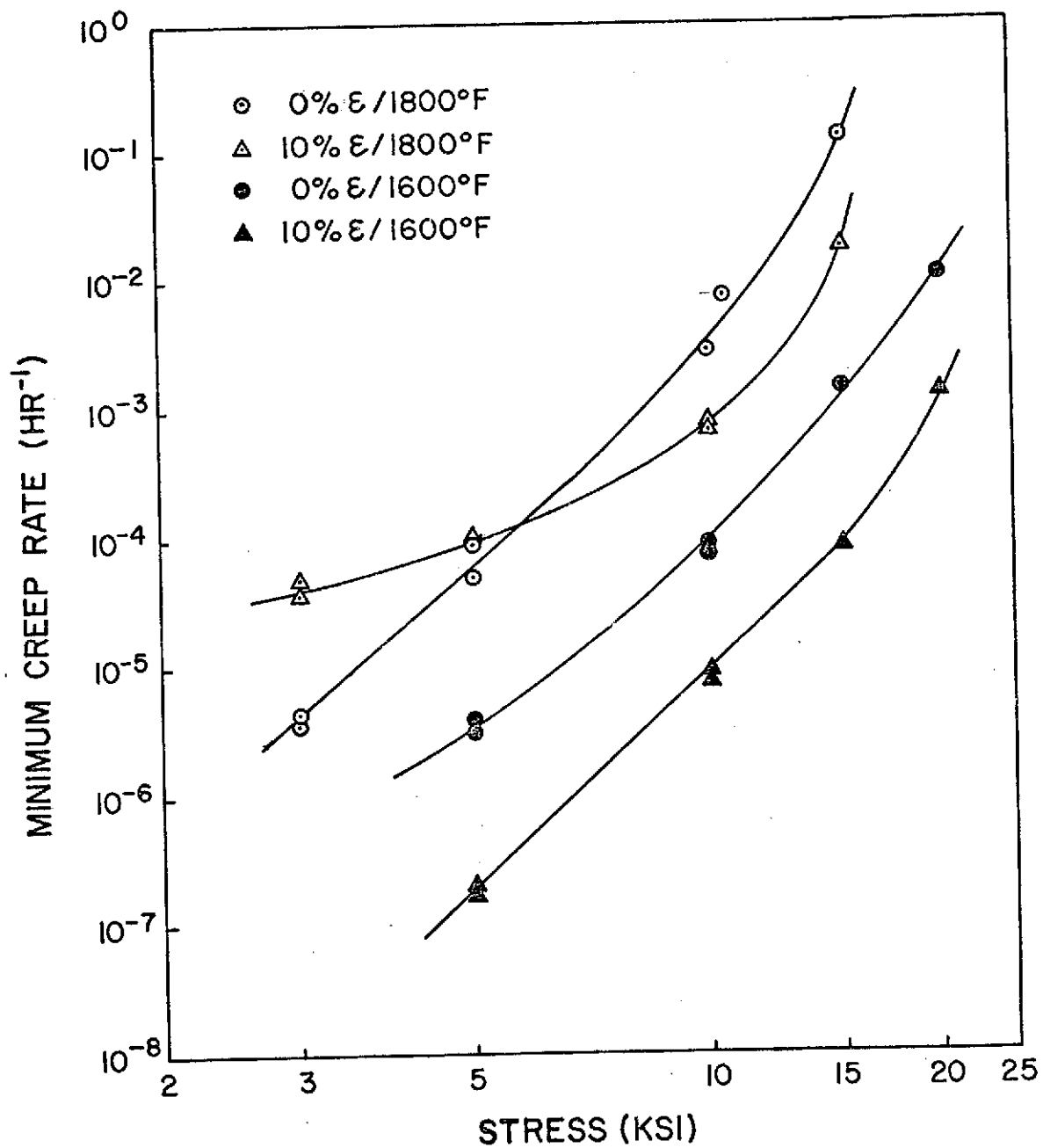


Figure 8. The effect of stress on the minimum creep rate of Haynes 188 alloy.

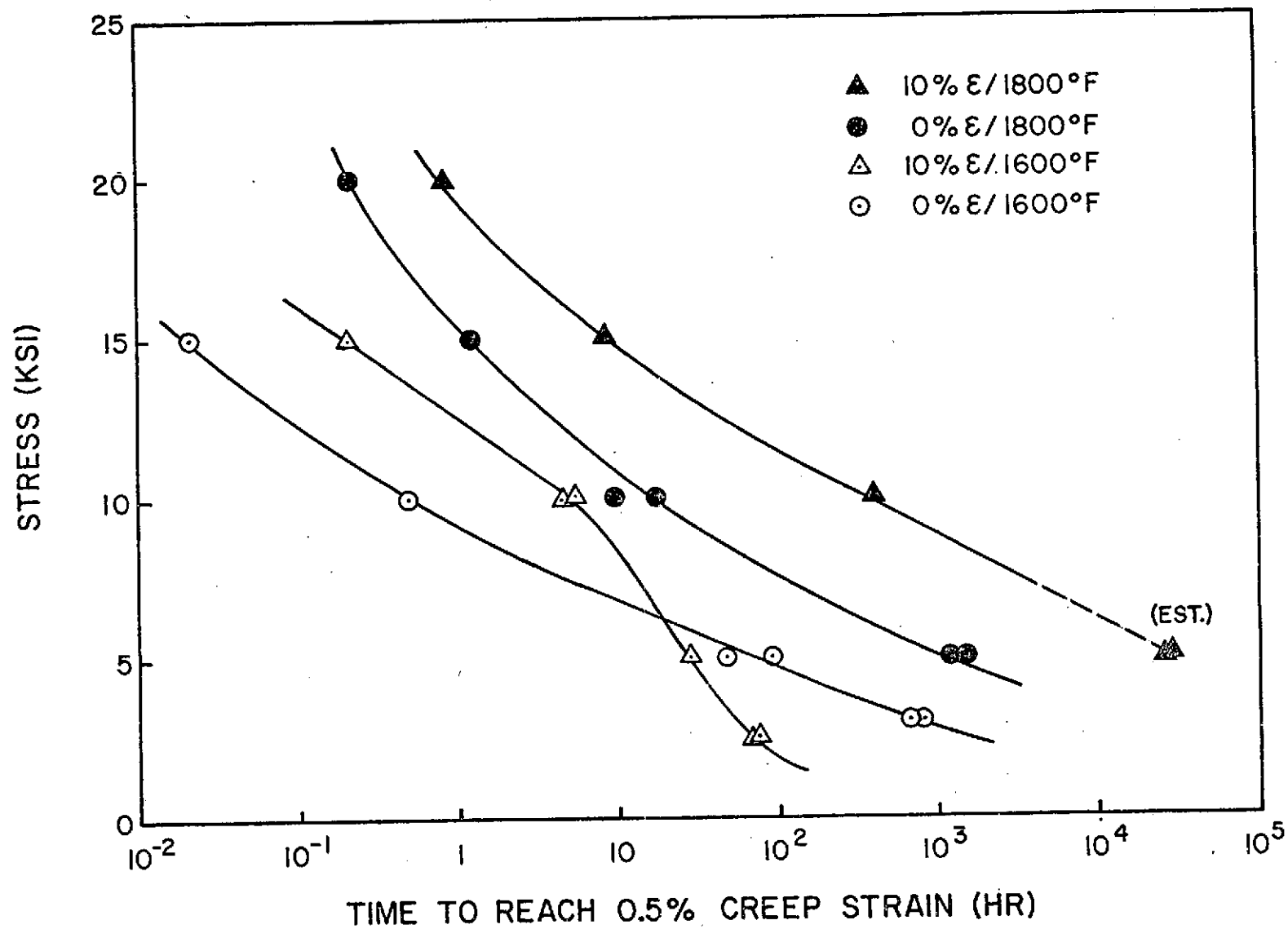


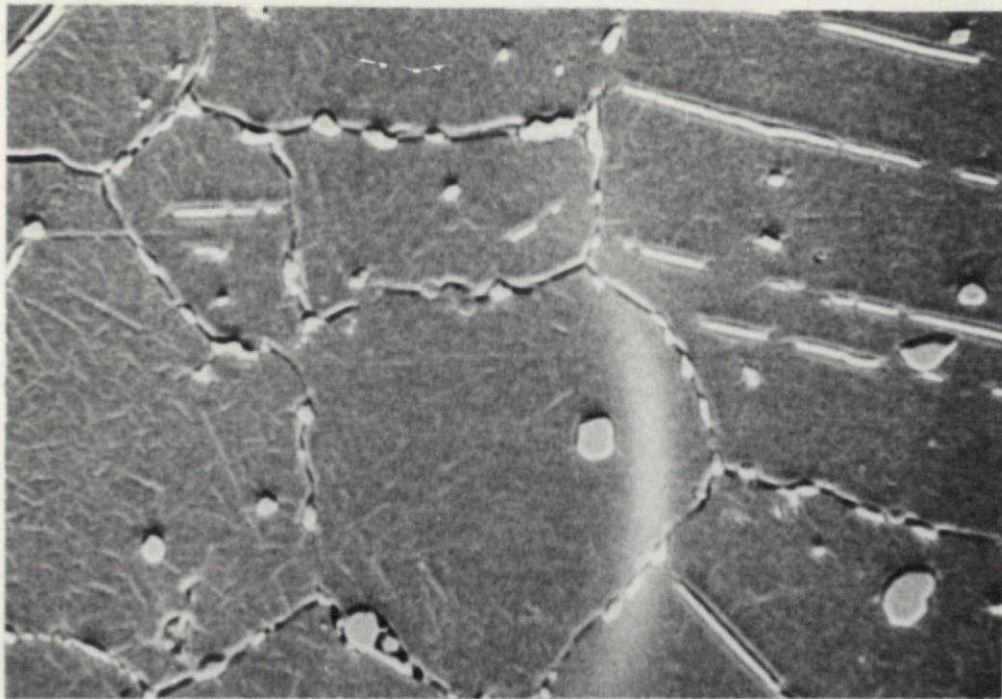
Figure 9. The effect of creep stress on the time to reach 0.5% creep strain for Haynes 188 alloy.

involving prior straining, it may be that the $M_{23}C_6$ carbide appears sooner and coalesces more rapidly to a distribution which provides poor creep resistance.

While definitive answers must await examination by transmission electron microscopy, some evidence of instability of grain structure has been obtained using scanning electron microscopy on polished and etched surfaces after creep (see Figure 10). Figure 10a shows the grain and carbide structure near the fracture zone for an as-received and preoxidized specimen crept at 1800°F and 5 ksi. Note the carbides at the grain boundaries and the tendency for some void formation there. Figure 10b shows the structure for a specimen prestrained 10%, preoxidized, and then crept at 1800°F and 5 ksi. Note that considerable grain boundary migration has occurred, and many carbides no longer reside at grain boundaries. There is also a corresponding decrease in grain boundary void formation, undoubtedly contributing to the increased creep ductility obtained. It may be that either: (1) the M_6C carbide is rapidly "over-aged" at 1800°F by enhanced kinetics due to the prestrain treatment, or (2) the $M_{23}C_6$ carbide is produced and coalesces rapidly. Further examination by electron microscopy will be required to clarify this point.

Reference to Figure 8 shows that, except for the low stresses at 1800°F where structural instability has been demonstrated, the effect of stress on minimum creep rate for Haynes 188 alloy can be represented by a power law with a stress exponent of about 5.5 for stresses below 10-15 ksi. This is about normal for Class II alloys. (1,3)

It is interesting to note that it has been found possible to correlate true creep fracture strain with a structural parameter devised from the relation (1,2):



(a) As-received and preoxidized.



(b) Prestrained 10% and preoxidized.

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Figure 10. Scanning electron micrographs of Haynes 188 alloy after creep at 1800°F and 5 ksi. 1500X.

$$\dot{\epsilon}_{\min} = S \left(\frac{\sigma}{E} \right)^n \exp (-\Delta H/RT)$$

where

- $\dot{\epsilon}_{\min}$ = the minimum creep rate,
- σ = the creep stress
- E = Young's modulus,
- ΔH = the activation energy for creep,
- R = the gas constant,
- T = the absolute temperature,
- n = the stress exponent, and
- S = a structural parameter.

By noting that S is proportional to $\dot{\epsilon}_{\min}/\sigma^n$ at a given temperature, and that fracture strain is a structural feature, a plot of $\dot{\epsilon}_{\min}/\sigma^5$ vs. true creep strain at fracture was made and is shown in Figure 11. The correlation is surprisingly good, considering that fracture strains are plotted for all tests, both as-received and thermomechanically treated. The correlation suggests that, whenever decreases in minimum creep rate are achieved by structural rearrangement in this alloy, a corresponding decrease in creep ductility will occur. The decrease in ductility, however, is no greater than would be obtained when decreasing the stress.

Transmission Electron Microscopy

The application of transmission electron microscopy was needed in the present study so that the observed creep behavior of the materials investigated can be interpreted in terms of dislocation and precipitate structure. The thinning technique developed by Dubose and Stiegler⁽¹²⁾ at Oak Ridge National Laboratory has been employed. This technique is quite rapid as compared with the standard methods, and it does not require the close attention of

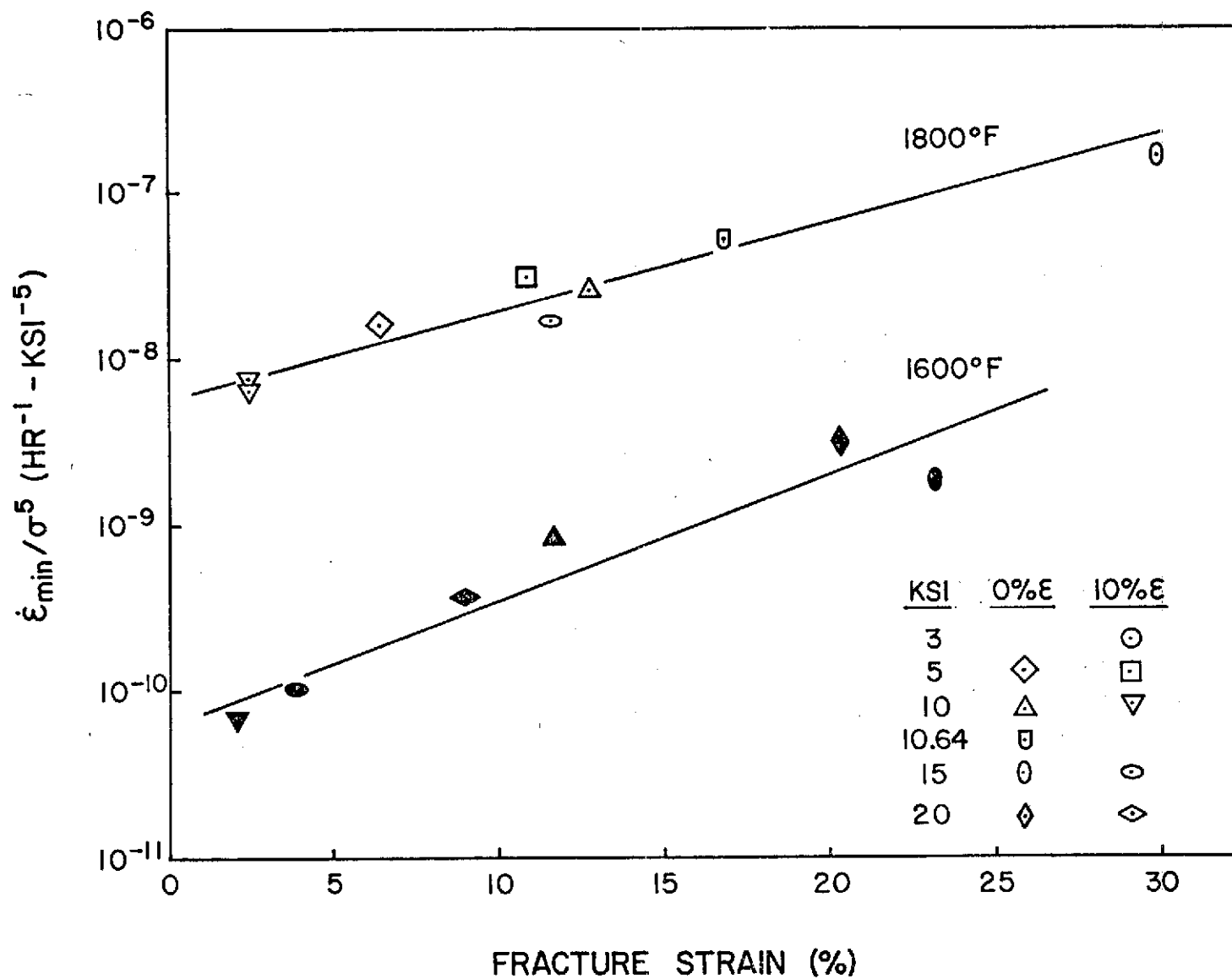


Figure 11. Correlation of creep fracture strain with minimum creep rate and stress for Haynes 188 alloy.

the operator. The technique consists of three separate operations. First, a wafer about 3mm in diameter by approximately 20 mils thick is cut from the bulk specimen using an electron discharge machine. Next, a jet of electrolyte is used to produce flat-bottom dimples from both sides of the disc leaving an area about 2 mils thick surrounded by a thick ring of unpolished material. Finally, the dimpled disc specimen is placed in a standard electropolishing dish where it is illuminated from one side by a focused light beam transmitted by a fiber-optics system. When a perforated hole is formed during polishing, the transmitted light activates a photo-cell detector which stops the polishing action.

The thinning apparatus which basically includes a jet polisher, a standard polishing dish, a semi-automatic cooling system, a D-C power supply up to 600 volts output capacity, and a light source and controller has been installed at Langley Research Center. A circulating ethylene glycol solution which can be cooled to -35°C by liquid nitrogen is used to cool the electrolytes in both thinning stages. Low operating temperature in the final stage of polishing is normally desired in order to eliminate any structural modification during thinning. This is especially true for titanium in which the formation of hydride precipitates can be prevented if temperature is kept below $\sim 15^{\circ}\text{C}^{(13)}$. Low temperature is also desired so that large uniform thin foils transparent to electrons can be more frequently obtained.

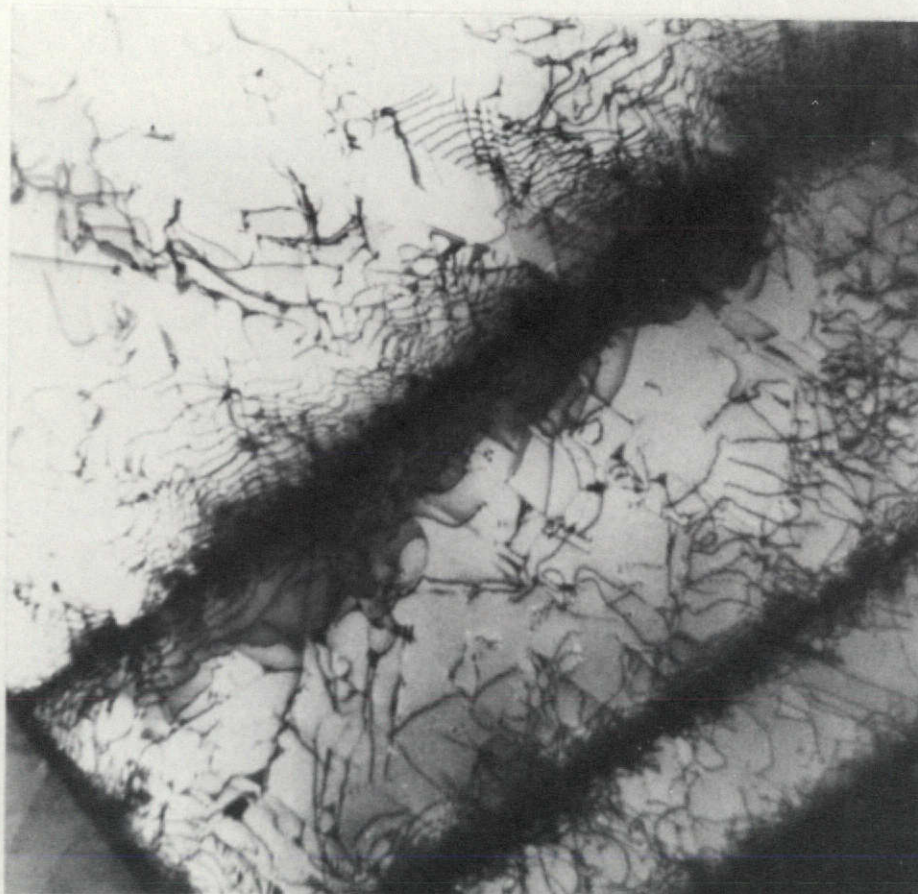
An electrolyte containing 33 cc. concentrated HNO_3 and 67 cc. methyl alcohol operating at room temperature and 300 milliamp was found to produce the best dimple configuration in Haynes 188 compared with the results obtained from several other solutions tested. However, the dimples so generated were not flat enough for final polishing. It was found later that the dimple configuration could be improved to a satisfactory shape if the operating

temperature was lowered to about 0° to 5°C . The same solution used in jet polishing was also found to be satisfactory for use in the final stage of thinning.

Although many weeks of experimentation were required to discover the conditions for producing reasonable thin foils of Haynes 188, this process has now been completed, and successful thinning operations are now possible for both the as-received and as-crept conditions. Examples of the recent successful foils are given in Figures 12 and 13. Figure 12 shows the as-received, mill annealed and stretch flattened condition. Note that considerable dislocation density is found near the twin boundaries, as previously suspected in discussion of Figure 2. Note that some stacking faults are visible. Figure 13 shows how the preoxidation treatment (1800°F , 1 hr) has decorated the twin boundaries with precipitate as also detected by scanning electron microscopy in Figure 3b. The dislocation density has also been reduced by the preoxidation treatment. Now that thinning techniques are available, the study of the changes in dislocation structure occurring in Haynes 188 during creep is being initiated.

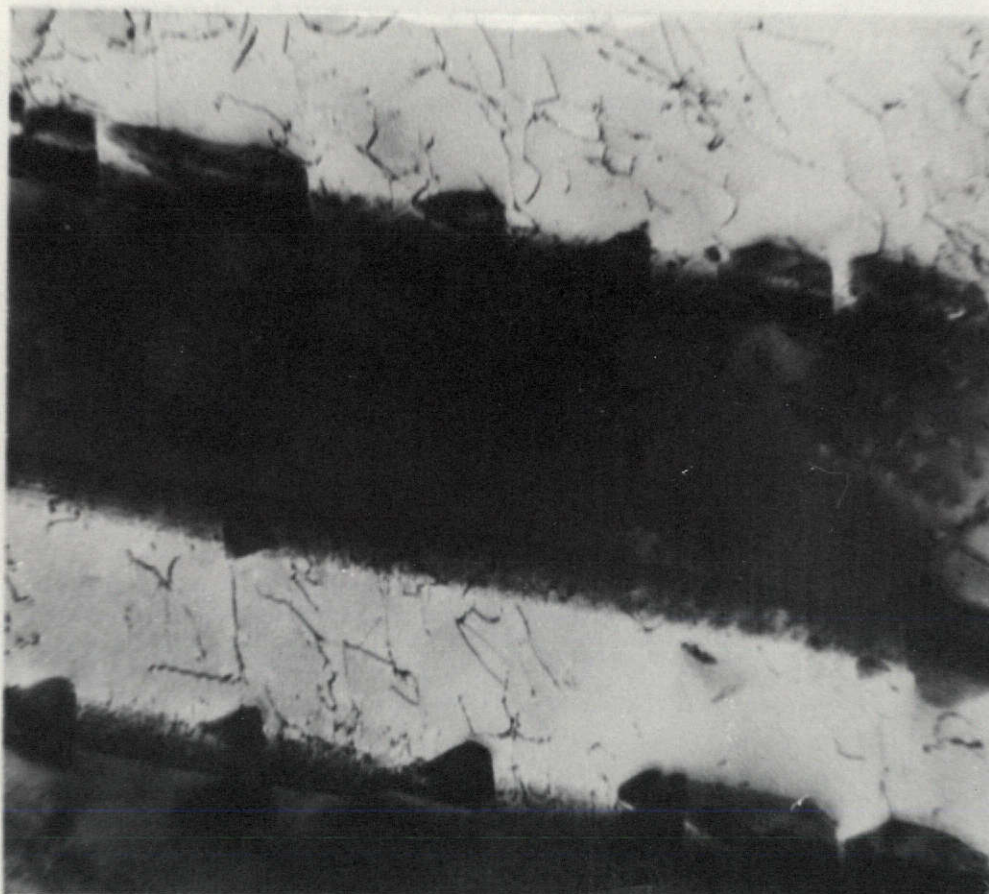
Computer Generation of Kikuchi Projections

In the present study, Kikuchi patterns (K-patterns) will be needed to determine the misorientation angles and axes between subgrains developed during creep tests. The normal trial and error procedures used in solving K-patterns are time consuming, especially for non-cubic crystals such as titanium. Two computer programs have been developed to provide for stereographic Kikuchi projections (K-projections) for FCC, BCC, diamond cubic, and HCP crystals for the purpose of identification of observed K-patterns⁽⁶⁾. A computer program has also developed to facilitate rapid and accurate determination of subgrain misorientation parameters⁽⁷⁾.



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Figure 12. Transmission electron micrograph showing as-received, stretch-flattened condition for Haynes 188 alloy. 20,000X.



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Figure 13. Transmission electron micrograph showing dislocation and precipitate structure of Haynes 188 alloy after preoxidation treatment for one hour at 1800°F. 28,000X

Using the two programs previously written, a set of stereographic projections could be obtained for any specified orientation and any desired projection sphere radius. The projection set included a K-projection, an identification zone projection, and a computer listing which was used to assist in indexing the Kikuchi lines in the K-projection. The listing, however, was still inconvenient. To solve this problem, a generalized new program has been developed to provide for K-projections for all the crystal systems. In this new program, the indices of each Kikuchi pair were plotted at the outskirts of each K-projection so that Kikuchi lines could be immediately identified. In addition, options were made available to plot standard stereographic zone axis projections, standard stereographic plane normal projections, and Wulff nets of any desired orientation and projection sphere radius. As an example, a K-projection for Ti at 100 KV for the $[\bar{1}2\bar{1}3]$ axis is shown in Figure 14 using a projection sphere radius of 25 cm. With the help of such projections, indexing of an observed K-pattern becomes a matter of simple comparison, and the tedious, and sometimes erroneous, aspects of pattern solution are minimized. This is particularly helpful in working with titanium and titanium alloys which contain the HCP α -phase.

Titanium and Ti-6Al-4V

Since no equipment for performing constant stress creep tests in a high vacuum environment was available for work on titanium, it was proposed that the components needed be purchased and that a new high vacuum constant stress creep machine be designed and built for this work at VPI&SU. Except for the aforementioned revisions of the computer program for Kikuchi analysis, progress to date on titanium and Ti-6Al-4V has been as follows:

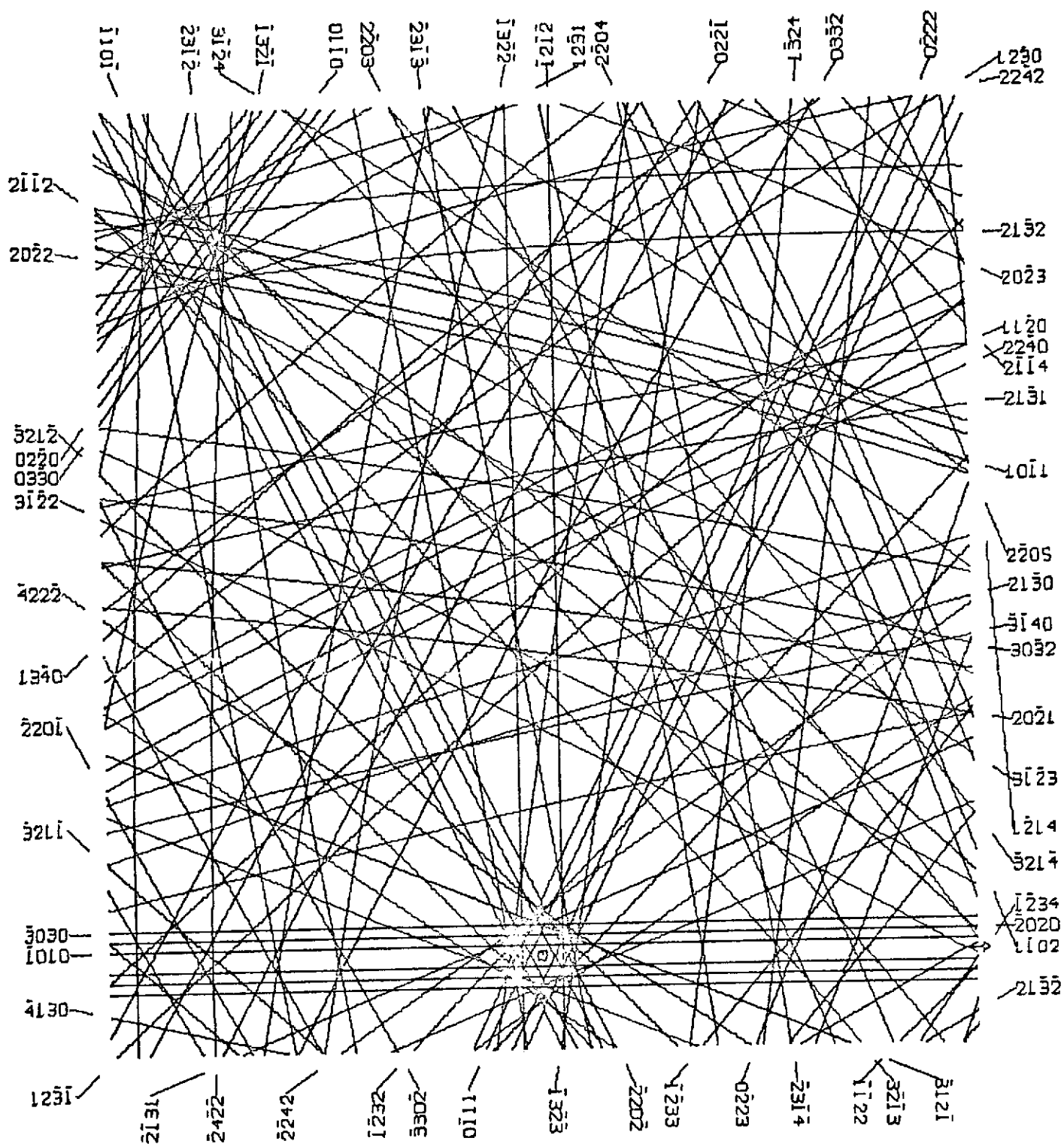


Figure 14. Kikuchi line projection for HCP α -Ti at 100kV with PSR = 25 cm. The improved program provides the line identifications shown at the edges.

1. A new constant-stress creep apparatus has been designed and is now nearing completion. This unit has the following characteristics: (1) specimen loads up to 250 lb, (2) creep testing at 10^{-6} Torr or better at temperatures up to 1900°F , (3) quick removal of the furnace from the specimen to provide for rapid cooling after creep to minimize microstructural changes, and (4) a parabolic lever arm which decreases the load during creep to maintain constant stress, beginning from a 5:1 lever ratio.

2. Equipment needed for the creep work has been purchased and has now been delivered. The equipment purchases include: (1) a Marshall creep furnace, (2) ionization and thermocouple vacuum gauge controller, (3) 15 cfm mechanical pump, (4) four-inch oil diffusion pump, and (5) appropriate high vacuum valves.

3. Glass apparatus needed for preparation of titanium thin foils according to the techniques of Stiegler and Dubose⁽¹²⁾ has been constructed and is now ready for use at VPI&SU. This apparatus is essentially identical to that now being used at NASA-Langley on the Haynes 188 alloy.

IV. PROJECT PERSONNEL

Principal Investigators

The research was conducted by C. T. Young and J. L. Lytton as co-principal investigators. Dr. Young is a Research Associate and Dr. Lytton is a Professor of Metallurgical Engineering at Virginia Polytechnic Institute and State University. Dr. Young is located at NASA-Langley during the course of this research and is responsible for performing creep tests and related microstructural examinations of Haynes 188 and Ti-6Al-4V alloys. Dr. Lytton participates in the analysis of the results and their relation to creep behavior and variations in processing variables, and is responsible for conducting the study of alpha-titanium and a portion of the work on Ti-6Al-4V at Virginia Polytechnic Institute. The work at VPI&SU requires the use of a Graduate Research Assistant, William Hochella. Dr. Young devotes full time to this project. Dr. Lytton has expended at least 25% of his time during the academic year and full time during 10 weeks of the summer period.

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